

# Document made available under the Patent Cooperation Treaty (PCT)

International application number: PCT/US2005/045133

International filing date: 13 December 2005 (13.12.2005)

Document type: Certified copy of priority document

Document details: Country/Office: US  
Number: 60/642,746  
Filing date: 10 January 2005 (10.01.2005)

Date of receipt at the International Bureau: 27 February 2006 (27.02.2006)

Remark: Priority document submitted or transmitted to the International Bureau in compliance with Rule 17.1(a) or (b)



World Intellectual Property Organization (WIPO) - Geneva, Switzerland  
Organisation Mondiale de la Propriété Intellectuelle (OMPI) - Genève, Suisse

1430467

# THE UNITED STATES OF AMERICA

TO ALL TO WHOM THESE PRESENTS SHALL COME:

UNITED STATES DEPARTMENT OF COMMERCE

United States Patent and Trademark Office

*February 20, 2006*

**THIS IS TO CERTIFY THAT ANNEXED HERETO IS A TRUE COPY FROM THE RECORDS OF THE UNITED STATES PATENT AND TRADEMARK OFFICE OF THOSE PAPERS OF THE BELOW IDENTIFIED PATENT APPLICATION THAT MET THE REQUIREMENTS TO BE GRANTED A FILING DATE.**

**APPLICATION NUMBER: 60/642,746**

**FILING DATE: *January 10, 2005***

**RELATED PCT APPLICATION NUMBER: *PCT/US05/45133***

**THE COUNTRY CODE AND NUMBER OF YOUR PRIORITY APPLICATION, TO BE USED FOR FILING ABROAD UNDER THE PARIS CONVENTION, IS *US60/642,746***



Certified by

Under Secretary of Commerce  
for Intellectual Property  
and Director of the United States  
Patent and Trademark Office

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

### PROVISIONAL APPLICATION FOR PATENT COVER SHEET

This is a request for filing a PROVISIONAL APPLICATION FOR PATENT under 37 CFR 1.53(c).

Express Mail Label No. EO 906 965 127 US

#### INVENTOR(S)

Given Name (first and middle [if any])	Family Name or Surname	Residence (City and either State or Foreign Country)
Daniel W.	Mayer	Wyoming, MN
Timothy A.	Ascherman	Elk River, MN

Additional inventors are being named on the \_\_\_\_\_ separately numbered sheets attached hereto

#### TITLE OF THE INVENTION (500 characters max):

**INSTRUMENT AND METHOD FOR DETECTING LEAKS IN HERMETICALLY SEALED PACKAGING**

Direct all correspondence to:

#### CORRESPONDENCE ADDRESS

☒ The address corresponding to Customer Number:

23403

OR

☐ Firm or  
Individual Name

Address

City

State

Zip

Country

Telephone

Fax

#### ENCLOSED APPLICATION PARTS (check all that apply)

☐ Application Data Sheet. See 37 CFR 1.76

☐ CD(s), Number of CDs \_\_\_\_\_

☒ Specification Number of Pages 11

☒ Other (specify) Postcard

☒ Drawing(s) Number of Sheets 1

**Application Size Fee:** If the specification and drawings exceed 100 sheets of paper, the application size fee due is \$250 (\$125 for small entity) for each additional 50 sheets or fraction thereof. See 35 U.S.C. 41(a)(1)(G) and 37 CFR 1.16(s).

#### METHOD OF PAYMENT OF FILING FEES AND APPLICATION SIZE FEE FOR THIS PROVISIONAL APPLICATION FOR PATENT

☒ Applicant claims small entity status. See 37 CFR 1.27.

TOTAL FEE AMOUNT (\$)

☐ A check or money order is enclosed to cover the filing fee and application size fee (if applicable).

100.00

☒ Payment by credit card. Form PTO-2038 is attached

☐ The Director is hereby authorized to charge the filing fee and application size fee (if applicable) or credit any overpayment to Deposit  
Account Number: \_\_\_\_\_ A duplicative copy of this form is enclosed for fee processing.

The invention was made by an agency of the United States Government or under a contract with an agency of the United States Government.

☒ No.

☐ Yes, the name of the U.S. Government agency and the Government contract number are: \_\_\_\_\_

SIGNATURE

*M. Sherrill*

Date

*10 Jan 05*

TYPED or PRINTED NAME Michael S. Sherrill

REGISTRATION NO. 32,302

(if appropriate)

TELEPHONE 651-426-2400

Docket Number: MCN226USPT01

#### USE ONLY FOR FILING A PROVISIONAL APPLICATION FOR PATENT

This collection of information is required by 37 CFR 1.51. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 8 hours to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.

# **INSTRUMENT AND METHOD FOR DETECTING LEAKS IN HERMETICALLY SEALED PACKAGING**

## **FIELD OF INVENTION**

**[0001]** The invention relates to instrument and techniques for analyzing the oxygen concentration within hermetically sealed packaging, and detecting leaks in hermetically sealed packaging.

## **BACKGROUND**

**[0002]** Products susceptible to spoilage, such as processed foods, nuts and sliced fruits and vegetables, are often placed in hermetically sealed packaging which has been flushed with an inert gas, such as nitrogen or argon, to achieve an oxygen concentration within the packaging of less than about 3% and thereby prolong the shelf-life of the product. Such packaging is commonly known as controlled atmosphere packaging (CAP) or modified atmosphere packaging (MAP).

**[0003]** Insufficient flushing of the atmosphere within the packaging and/or leaks in the packaging can significantly reduce the anticipated shelf life, resulting in undesired spoilage. Hence, proper quality control efforts for CAP/MAP typically involve at least periodic testing of packaging to determine the oxygen content within the hermetically sealed packaging and the presence of any leaks in the packaging.

**[0004]** A variety of instruments and methods for analyzing the oxygen concentration within the headspace of CAP/MAP are known to those skilled in the art, including the PAC CHECK® series of oxygen headspace analyzers offered by MOCON, Inc. of Minneapolis, Minnesota. Briefly, these instruments involve puncturing a sample package with a hollow needle through which a sample of the headspace gases can be withdrawn for analysis by an oxygen sensor. See, United States Patent No. 5,212,993 issued to William Mayer, the disclosure of which is hereby incorporated by reference.

**[0005]** A variety of instruments and methods are also known for leak detection in hermetically sealed packaging. Leak detection typically involves the creation of a pressure differential between the pressure inside the packaging (internal pressure) and the pressure outside the packaging (external pressure) - such as by compressing the packaging or pulling a vacuum in the atmosphere surrounding the packaging, followed by the detection of any change in a given variable which could be correlated to the presence of a leak in the packaging. Specific examples include submersion of the packaging into a liquid and detecting any liquid within the packaging (United States Patent No. 6,763,702 issued to Allen Chien et al.), squeezing of the packaging and detecting of any pressure decay (United States Patent No. 6,427,524 issued to Frank Raspante et al.), squeezing the packaging and detecting any volume decay (United States Patent No. 5,533,385 issued to William Frievalt) and placing the packaging within a vacuum chamber and detecting any loss of vacuum (United States Patent. No. 5,150,605 issued to Edwin Simpson).

**[0006]** While each of these instruments and techniques are generally effective for analyzing the oxygen concentration within hermetically sealed packaging, or detecting leaks in hermetically sealed packaging, a substantial need continues to exist for (i) a quick, easy, inexpensive and accurate instrument and technique for detecting leaks in hermetically sealed packaging, and (ii) an instrument and technique capable of providing both an accurate analysis of the oxygen concentration within hermetically sealed packaging and the detection of any leaks in the hermetically sealed packaging using the same individual package and using a single integrated instrument to conduct both tests.

## **OUTLINE OF THE INVENTION**

**[0007]** A first aspect of the invention is an instrument for detecting leaks in hermetically sealed packaging. The instrument includes (a) a needle having a lumen, (b) a mass flow rate sensor in sealed fluid communication with the lumen defined by the needle, and (c) a vacuum pump which is in fluid communication with the lumen defined by the needle for evacuating gas from the hermetically sealed packaging, and in fluid communication with the mass flow rate sensor for directing mass flow from the evacuated packaging into operable contact with the mass

flow rate sensor so as to permit sensing of any continuing mass flow from the evacuated packaging.

**[0008]** A second aspect of the invention is an instrument for analyzing oxygen concentration of a gas within hermetically sealed packaging and detecting leaks in the hermetically sealed packaging. The instrument includes (a) a needle having a lumen, (b) an oxygen sensor in sealed fluid communication with the lumen defined by the needle, (c) a mass flow rate sensor in sealed fluid communication with the lumen defined by the needle, and (d) a vacuum pump which is (i) in fluid communication with the lumen defined by the needle for (A) pumping a sample of a gas from within a hermetically sealed packaging through the lumen of the needle and into operable contact with the oxygen sensor for permitting sensing of an oxygen concentration of the sample, and (B) evacuating the gaseous content of the hermetically sealed packaging, and (ii) in fluid communication with the mass flow rate sensor for directing mass flow from the evacuated packaging into operable contact with the mass flow rate sensor so as to permit sensing of any continuing mass flow from the evacuated packaging.

**[0009]** A third aspect of the invention is a method of detecting leaks in hermetically sealed packaging. The method involves (a) selecting a hermetically sealed packaging, (b) puncturing the hermetically sealed packaging with a hollow needle having a lumen, (c) evacuating any gaseous content from within the hermetically sealed packaging through the lumen of the needle to form a vacuum within the hermetically sealed packaging, and (d) measuring mass flow rate from within the evacuated hermetically sealed packaging, whereby a mass flow rate from the evacuated hermetically sealed packaging above a threshold value indicates a leak in the hermetically sealed packaging.

**[0010]** A fourth aspect of the invention is a method of analyzing oxygen concentration of a gas within hermetically sealed packaging and detecting leaks in the hermetically sealed packaging. The method involves (a) selecting a hermetically sealed packaging, (b) puncturing the hermetically sealed packaging with a hollow needle having a lumen, (c) pumping a sample of the gas within the hermetically sealed packaging through the lumen of the needle and into operable contact with an oxygen sensor for sensing the oxygen concentration in the sample, (d)

evacuating the gaseous content from within the hermetically sealed packaging through the lumen of the needle to form a vacuum within the hermetically sealed packaging, and (e) measuring mass flow rate from the evacuated hermetically sealed packaging, whereby a mass flow rate from the evacuated hermetically sealed packaging above a threshold value indicates a leak in the hermetically sealed packaging.

## **SUMMARY OF THE INVENTION**

**[0011]** A first aspect of the invention is an instrument for detecting leaks in hermetically sealed packaging. The instrument includes (a) a needle having a lumen, (b) a mass flow rate sensor, and (c) a vacuum pump. The mass flow rate sensor is in sealed fluid communication with the lumen defined by the needle. The vacuum pump is in fluid communication with both the lumen defined by the needle and the mass flow rate sensor for evacuating gas from the hermetically sealed packaging and allow the mass flow rate sensor to sense any continuing mass flow from the evacuated packaging.

**[0012]** A second aspect of the invention is an instrument for analyzing oxygen concentration of a gas within hermetically sealed packaging and detecting leaks in the hermetically sealed packaging. The instrument includes (a) a needle having a lumen, (b) an oxygen sensor, (c) a mass flow rate sensor, and (d) a vacuum pump. The oxygen sensor and the mass flow rate sensor are in sealed fluid communication with the lumen defined by the needle. The vacuum pump is in fluid communication with the lumen defined by the needle and both the oxygen sensor and the mass flow rate sensor for (A) pumping a sample of a gas from within a hermetically sealed packaging through the lumen of the needle and into operable contact with the oxygen sensor for permitting sensing of an oxygen concentration of the sample, (B) evacuating the gaseous content of the hermetically sealed packaging, and (C) allow the mass flow rate sensor to sense any continuing mass flow from the evacuated packaging.

**[0013]** A third aspect of the invention is a method of detecting leaks in hermetically sealed packaging. The method involves (a) selecting a hermetically sealed packaging, (b) puncturing the hermetically sealed packaging with a hollow needle having a lumen, (c) evacuating any

gaseous content from within the hermetically sealed packaging through the lumen of the needle to form a vacuum within the hermetically sealed packaging, and (d) measuring mass flow rate from within the evacuated hermetically sealed packaging. Sensing of a mass flow rate from the evacuated hermetically sealed packaging above a threshold value indicates a leak in the hermetically sealed packaging.

**[0014]** A fourth aspect of the invention is a method of analyzing oxygen concentration of a gas within hermetically sealed packaging and detecting leaks in the hermetically sealed packaging. The method involves (a) selecting a hermetically sealed packaging, (b) puncturing the hermetically sealed packaging with a hollow needle having a lumen, (c) pumping a sample of the gas within the hermetically sealed packaging through the lumen of the needle and into operable contact with an oxygen sensor for sensing the oxygen concentration in the sample, (d) evacuating the gaseous content from within the hermetically sealed packaging through the lumen of the needle to form a vacuum within the hermetically sealed packaging, and (e) measuring mass flow rate from the evacuated hermetically sealed packaging. Sensing of a mass flow rate from the evacuated hermetically sealed packaging above a threshold value indicates a leak in the hermetically sealed packaging.

#### **BRIEF DESCRIPTION OF THE DRAWINGS**

**[0015]** Figure 1 is a side view of one embodiment of the invention.

**[0016]** Figure 2 is an enlarged cross-sectional side view of the distal end of the needle shown in FIG 1.



**DETAILED DESCRIPTION OF THE INVENTION**  
**INCLUDING A BEST MODE**

***Nomenclature***

<b>10</b>	Instrument
<b>20</b>	Needle
<b>21</b>	Distal End of Needle
<b>29</b>	Lumen of Needle
<b>30</b>	Pressure Sensor
<b>40</b>	Oxygen Sensor
<b>50</b>	Valve
<b>60</b>	Vacuum Pump
<b>70</b>	Mass Flow Rate Sensor
<b>80</b>	Collective Reference to Tubing 80a, 80b and 80c
<b>80a</b>	Length of Tubing Interconnecting the Needle and the Oxygen Sensor
<b>80b</b>	Length of Tubing Interconnecting the Oxygen Sensor and the Vacuum Pump
<b>80c</b>	Length of Tubing Interconnecting the Vacuum Pump and the Mass Flow Rate Sensor
<b>90</b>	Vent
<b>100</b>	Packaging
<b>101</b>	Solids Content of Packaging
<b>102</b>	Gaseous Content of Packaging
<b>200</b>	Septum

***Structure***

**[0017]** As shown in FIG. 1, a first aspect of the invention is an instrument **10** for analyzing the oxygen concentration of the gaseous content **102** of hermetically sealed packaging **100** and detecting leaks in the hermetically sealed packaging **100**.

[0018] The instrument 10 can be effectively employed with a wide variety of hermetically sealed packaging 100 ranging from rigid packaging such as poly vinyl chloride tubes, through semi-flexible packaging such as wax-coated cartons and thin-walled polyethylene bottles, to flexible packaging such as bags made from polyethylene terephthalate (*i.e.*, MYLAR®) or polyethylene films.

[0019] Referring to FIG. 1, the first aspect of the instrument 10 includes a needle 20, a pressure sensor 30, an oxygen sensor 40, an optional valve 50, a vacuum pump 60, a mass flow rate sensor 70, and appropriate tubing 80a, 80b and 80c (hereinafter collectively referenced as tubing 80) for sealingly interconnecting the various components of the instrument 10.

[0020] As shown in FIG. 2, the needle 20 is configured and arranged with a lumen 29 and a sharp pointed distal end 21 effective for piercing a sidewall (unnumbered) of the packaging 100. A septum 200 is preferably adhered to the sidewall of the packaging 100 - especially when the packaging 100 is highly flexible - prior to piercing of the packaging 100 with the needle 20 in order to maintain a hermetic seal around the needle 20.

[0021] Referring to FIG. 1, a vacuum pump 60 is sealingly connected to the lumen 29 of the needle 20 by tubing 80. Once the needle 20 has been inserted through packaging 100, operation of the vacuum pump 60 is effective for pulling a sampling of the gaseous content 102 of the packaging 100 out from the packaging 100 through the lumen 29 of the needle 20 and into operable contact with an oxygen sensor 40.

[0022] Substantially any type of vacuum pump 60 is suitable for use in the instrument 10, with selection dependent primarily upon choice of power source (*i.e.*, battery or electrical power lines), desired level of portability (*i.e.*, hand-held or desk-top), and intended use (*i.e.*, testing of large volume or small volume packaging). For most applications, a vacuum pump 60 with a maximum gas volumetric flow rate of about 250 to 1,000 cm<sup>3</sup>/minute and capable of pulling a maximum vacuum of about 1-15 lb/in<sup>2</sup>, preferably 4-8 lb/in<sup>2</sup>, using standard consumer batteries (*e.g.*, AAA, AA, A, C, D or 9-volt batteries) will be sufficient.

[0023] The oxygen sensor **40** may be selected from any of a wide variety of oxygen sensors readily available from a number of sources, such as the PAC CHECK® series of oxygen headspace analyzers offered by MOCON, Inc. of Minneapolis, Minnesota. The oxygen sensor **40** is preferably positioned upstream from the vacuum pump **60** to avoid potential contamination of the gas sample taken from the packaging **100** for analysis.

[0024] As described in detail in United States Patent No. 5,212,993, the disclosure of which is hereby incorporated by reference, a valve **50**, such as a 3-way valve, is preferably positioned between the oxygen sensor **40** and the vacuum pump **60** for permitting the pressure of a gas sample pulled from the packaging **100** into the oxygen sensor **40** by the pump **60** to be normalized prior to analysis. Such normalization of the gas sample pressure has been found to enhance the accuracy of the analysis.

[0025] A mass flow rate sensor **70** is positioned downstream from the vacuum pump **60** for measuring the mass flow rate of the gaseous content **102** pulled from packaging **100** by the vacuum pump **60**. Alternatively, the mass flow rate sensor **70** may be positioned upstream from the vacuum pump **60**. Of interest is the mass flow rate measured by the mass flow rate sensor **70** after the gaseous content **102** has been evacuated from the packaging **100** by the vacuum pump **60** and a steady state vacuum has been established within the packaging **100**. The mass flow rate measured at this stage is indicative of the presence of a leak in the packaging **100** when the mass flow rate is greater than or equal to a threshold value and indicative of the absence of any leaks in the packaging **100** when the mass flow rate is less than the threshold value.

[0026] Selection of a practical threshold value depends upon a number of factors including the level of the vacuum pulled by the vacuum pump **60**, the material from which the packaging **100** is constructed, etc. Generally, a threshold value of about 0.1 cm<sup>3</sup>/min may be effectively employed as the threshold value when the vacuum pulled by the vacuum pump **60** is between about 4-8 lb/in<sup>2</sup>.

[0027] Suitable gas mass flow rate sensors **70** for use in the instrument **10** are available from a number of sources, including MKS Instruments or Wilmington, Massachusetts.

[0028] A pressure sensor 30 is employed between the needle 20 and the vacuum pump 60, preferably between the needle 20 and the oxygen sensor 40, for measuring and reporting the pressure within the packaging 100 while the vacuum pump 60 is operating.

[0029] The pressure sensor 30, oxygen sensor 40, optional valve 50, vacuum pump 60, and mass flow rate sensor 70 are preferably operably interconnected through a microcontroller or processor (not shown) for controlling operation of the various components, and receiving and processing the data signals generated by the various sensors. These components, along with the associated tubing 80 and a power source (not shown), are preferably retained within a single housing (not shown) which is equipped with (i) an inlet port (not shown) configured and arranged to attach to a length of tubing 80a in order to place the needle 20 into fluid communication with the components retained within the housing, and (ii) a user input device (not shown) and a visual display panel (not shown) for communicating with a user.

[0030] The instrument 10 may be constructed as a portable or desktop unit.

### *Use*

[0031] A unit of packaging 100 having a solids content 101 and a gaseous content 102 is selected for analysis. A septum 200 is optionally adhered to the outer surface (unnumbered) of the packaging 100. The septum 200 and packaging 100 are perforated by the distal end 21 of the needle 20 a sufficient distance to place the lumen 29 into fluid communication with the interior contents of the packaging 100. The needle 20 is then left in the inserted position for the balance of the procedure. The balance of the procedure may be effected manually or automatically. The following disclosure shall be presented based upon automated operation of the instrument 10.

[0032] A user initiates analysis by pressing a START button (not shown). The valve 50 is actuated so as to place the needle 20 into fluid communication with the vacuum pump 60 and close the vent 90. Vacuum pump 60 is then activated to pull a sample of the gaseous content 102 from the packaging 100 into the oxygen sensor 40 through the length of tubing 80a. Typically a

sample of about 2-5 ml is sufficient. The vacuum pump **60** is then deactivated, the valve **50** actuated to open the vent **90** and allow the pressure of the gaseous sample within the oxygen sensor **40** to be normalized. Once the pressure of the gaseous sample is normalized the valve **50** is actuated to close the vent **90** and the oxygen sensor **40** is activated to sense, record and report the oxygen concentration in the gaseous sample withdrawn from the packaging **100**.

[0033] Upon completion of the analysis performed by the oxygen sensor **40**, the oxygen sensor **40** is deactivated and the vacuum pump **60** activated to evacuate the gaseous content **102** from the packaging **100** and pull a vacuum. The mass flow rate sensor **70** is activated and the mass flow rate through the tubing **80** is sensed after the gaseous content **102** from the packaging **100** has been evacuated. The sensed mass flow rate may simply be recorded and reported, but is preferably first compared to a threshold value and the sensed mass flow rate recorded and reported along with an indication of NO LEAK DETECTED when the mass flow rate at steady state vacuum is less than the threshold value, or an indication of LEAK DETECTED when the mass flow rate at steady state vacuum is greater than or equal to the threshold value. The vacuum pump **60** and mass flow rate sensor **70** are then deactivated.

## **ABSTRACT**

An instrument for detecting leaks in a hermetically sealed package. The instrument includes (i) a hollow needle, (ii) a mass flow rate sensor in sealed fluid communication with the lumen defined by the hollow needle, and (iii) a vacuum pump. The vacuum pump is in fluid communication with the lumen defined by the needle for evacuating gas from the hermetically sealed package, and in fluid communication with the mass flow rate sensor for directing mass flow from the evacuated package into operable contact with the mass flow rate sensor so as to permit sensing of any continuing mass flow from the evacuated package. The instrument may further include an oxygen sensor, also in sealed fluid communication with the lumen defined by the hollow needle, for analyzing the oxygen concentration of the gas within the hermetically sealed package. When an oxygen sensor is incorporated into the instrument, the vacuum pump is configured and arranged to pump a sample of the gas from within the hermetically sealed package into operable contact with the oxygen sensor.

